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IS 1448-72 (1968): Methods of test for petroleum and its products, Part 72: Volatility of liquefied petroleum gases [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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VOLATILITY OF LIQUEFIED PETROLEUM GASES

[P : 72]

(Second Reprint MAY 1988)

Adopted from ASTM Designation D 1837-64

1. SCOPE

1.1 This method is intended to determine the volatility of the various types of liquefied petroleum gases (LPG) as indicated by the approximate amount of higher boiling constituents present.

1.1.1 The test results, when properly related to the observed vapour pressures and densities of the product, may be used to indicate the approximate concentration of butanes in the propane type LPG, and pentanes in the butane-propane mixture type and butane type LPG. Presence of hydrocarbon compounds (less volatile than those of which the LPG product is primarily composed) is indicated by an increase in the temperature at the 95 percent evaporated point.

2. OUTLINE OF THE METHOD

2.1 The product is refrigerated by means of a cooling coil, and 100 ml of the liquid is collected in a weathering tube. The liquid is allowed to weather under specified conditions, and the temperature is observed when 95 percent of the initial quantity has evaporated. Correction for variation of barometric pressure is made.

3. APPARATUS

3.1 Weathering Tube — The tube (*see* Fig. 1) shall be made of glass and shall be of 100 ml capacity. It shall be cone shaped, conforming to the dimensions shown in Fig. 1 and shall be of 1·5 to 2 mm wall thickness to weigh not less than 60 g. Its graduations shall be distinct and shall read upward from the bottom of the tube. The values of the divisions for each range with limits of error for the concerned range are given in Table 1.

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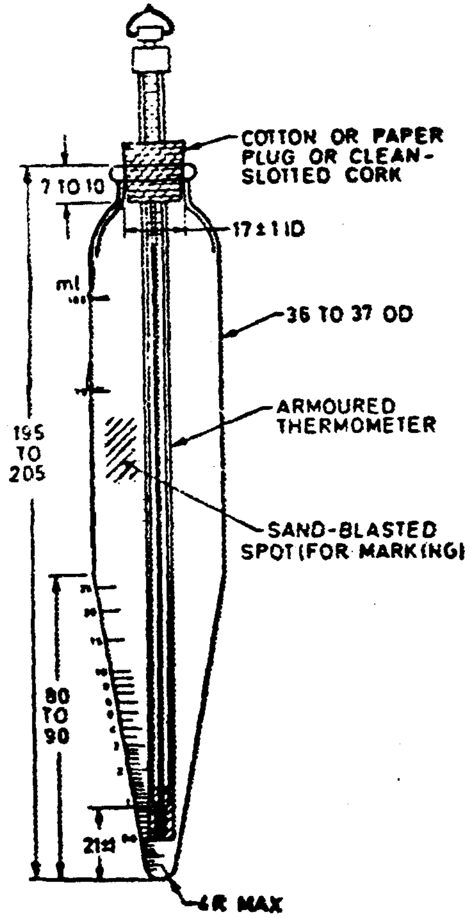

All dimensions in millimetres.

Fig. 1 Weathering Tube

3.1.1 The weathering tube shall contain an armoured thermometer as specified in **3.4** with its tip at a distance of 21 ± 1 mm from the inside bottom-edge of the tube as shown in Fig. 1.

TABLE 1 LIMITS OF ERROR FOR THE WEATHERING TUBE

(*Clause* 3.1)

Range (1) ml			Scale Division (2) ml	Limit of Error for the Range (3) ml	Numbered (4) ml
0	to	0·1	0·05	±0·02	—
0·1	,,	0·3	0·05	±0·03	—
0·3	,,	0·5	0·05	±0·05	0·5
0·5	,,	1·0	0·1	±0·05	1
1	,,	2	0·1	±0·1	2
2	,,	3	0·2	±0·1	3
3	,,	5	0·5	±0·2	4,5
5	,,	10	1·0	±0·5	6,8,10
10	,,	25	5·0	±1·0	15,20,25
25	,,	100	25·0	±1·0	50,75,100

3.2 Tube Support — Means shall be provided for supporting the weathering tube by its neck in a vertical position.

3.3 Water Bath (for Use in Tests on Butane and Butane-Propane Mixture Types of LPG Only) — a shallow container filled with clean water having a maintained temperature ranging from 15° to 21°C and a depth of 38 mm.

3.4 Thermometer* — an armoured thermometer with a suitable mercury-thallium alloy being used as actuating fluid and conforming to the following requirements:

Range	−48° to + 4°C
Immersion	35 mm
Graduation	0·2°C
Longer lines at each	1°C
Figured at each	2°C
Overall length	299 to 305 mm
Stem diameter	6·5 to 8·0 mm
Bulb shape	Cylindrical
Bulb length	15 to 20 mm
Bulb diameter	6 to 7 mm
Length of graduated portion	170 to 215 mm
Distance between bulb bottom and −48°C mark	50 to 75 mm
Expansion chamber	To allow heating to 66°C
Scale error	Not to exceed ± 0·2°C

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*Till such time a thermometer to these requirements is available, the thermometer No. 99F prescribed in ASTM Designation E-1 may be used and the observations converted to degrees Celsius from degrees Fahrenheit.

3.4.1 Do not remove the armour from the thermometer during the test. Place the bottom of the armour as low in the tube as possible, pipette 5 ml of water and observe the water level in the tube. The 5-percent residue readings are to be taken at the same level with the armoured thermometer in the same position.

3.5 Sample Pre-cooling Equipment

3.5.1 *Cooling Vessel* — any suitable wide mouthed metal container or Dewar flask at least 65 mm internal diameter and 300 mm deep.

3.5.2 *Cooling Coil* (*see* Fig. 2) — approximately 6 m of about 6 mm outer diameter soft copper tubing, wound around a hollow mandrel of about 55 mm diameter with adjacent turns touching. Run the lower end of the tube through the centre of the mandrel before winding so that the finished coil snugly fits inside the cooling vessel. When assembled, the top of the coil shall be at least 25 mm below the top of the cooling vessel and the open ends of the coil shall not be more than a few centimetres above. Connect the down stream end of the coil to a 3-mm needle valve having an outlet connection not more than 75 mm long.

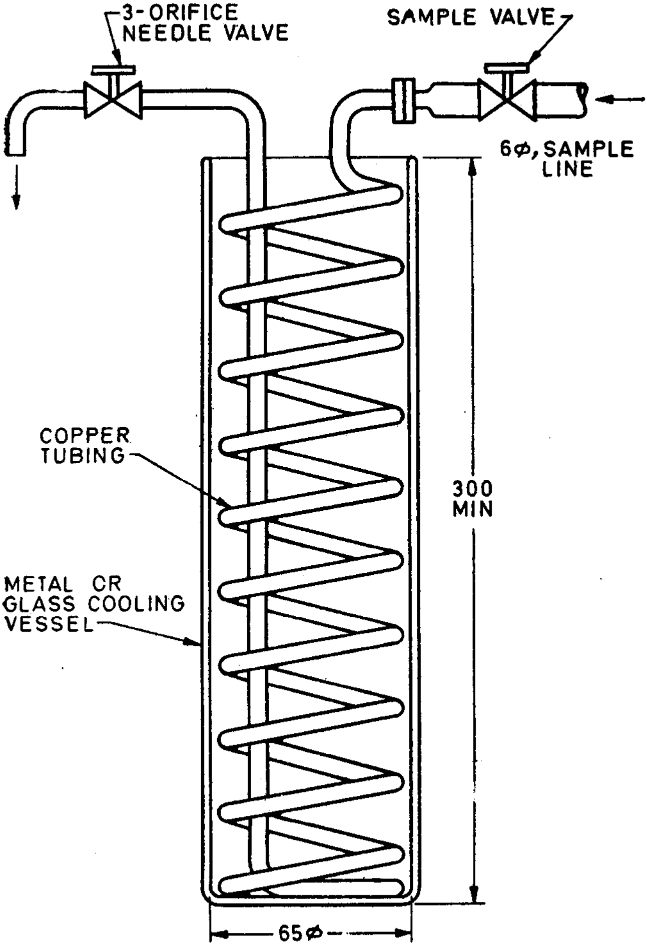
3.6 Pre-coolant — liquefied petroleum gas from the same container from which the sample is to be taken. Other refrigerants having a boiling point lower than the initial boiling point of the sample may also be used. Use a non-flammable pre-coolant where safety requires it.

4. PROCEDURE

4.1 Obtaining a Test Sample — Fill the cooling vessel with the pre-coolant so as to cover the cooling coil. Attach the inlet of the cooling coil to the source from which the sample is to be taken with a short line connection pipe of 6 mm diameter having a sampling valve large enough to prevent vaporization of the material due to the drop in pressure across the valve seat. Purge the sampling line and cooling coil by opening both the sampling valve and the 3-mm needle valve on the down-stream end of the cooling coil. Fill the weathering tube with the sample flowing through the cooling coil. Empty this first sample, add one or two grains of charcoal, and then refill the weathering tube to the 100-ml mark with fresh liquid sample passing through the cooling coil. Carefully insert into the centrifuge tube the pre-cooled armoured thermometer up to the desired depth (*see* **3.1.1**). Centre the armoured thermometer in the tube by means of a slotted cork.

4.2 Weathering of LPG

4.2.1 *Butane and Butane-Propane Mixture Types* — If the temperature of the sample is below −12°C, allow it to weather in the atmosphere until the temperature has reached −12°C. At this point, place the weathering tube



All dimensions in millimetres.

FIG. 2 SAMPLE PRE-COOLING EQUIPMENT

with the armoured thermometer still in place in the water-bath in a vertical position submerging it to the 1·5 ml mark and allow the contents to weather.

> **CAUTION :** *Safe means for disposal of products of weathering should be provided.*

4.2.2 *Propane Type* — Allow the sample to weather in the atmosphere taking care to disturb the frost on the tube as little as possible (*see* Caution in **4.2.1**). An acetone or alcohol swab may be used to remove frost sufficient to permit reading of the temperature.

4.3 Reading of Temperature — When the liquid level in the weathering tube, with the armoured thermometer in place, corresponds with the level previously determined after 5 ml of water was put in the weathering tube, read and record the temperature of the sample in accordance with **4.2.1** and **4.2.2** depending on the type of LPG sample.

4.4 Temperature Correction — Following the final temperature reading (*see* **4.3**), remove the armoured thermometer from the weathering tube and place it in a bath of finely crushed ice up to the immersion point. Observe the reading of the thermometer when a constant reading is obtained. If the thermometer reading is less than 0°C, read this difference; this shall be added to the final test reading. If the thermometer reading is more than 0°C, again read the difference; this difference shall be subtracted from the final test reading. Any thermometer which varies more than 0·6°C in either direction shall be discarded.

NOTE — A high reading of the thermometer when it is placed in ice usually indicates that there is a break in the mercury-thallium thread. This may be corrected by warming the thermometer gently in a warm water-bath to drive the break upward into the expansion chamber at the top of the thermometer. While the mercury-thallium is continuous in the upper chamber, tap the bottom of the thermometer on a hard but cushioned surface to join the liquid into a continuous thread. A low reading in ice usually indicates that some of the liquid has remained in the expansion chamber. To correct this, allow the thermometer to warm so that the liquid enters the chamber and tap as previously instructed.

5. CALCULATION AND REPORTING

5.1 Correct the thermometer reading at the 95 percent boiling point (5 percent residue) for the thermometer error.

5.2 Correct further the value obtained in **5.1** from the observed atmospheric pressure in millimetres of mercury to a base barometric pressure of 760 mm as given below:

Weathering Test (for LPG Type)	*Correction for Each 10 mm of Mercury (for Pressure)*	
	Above 760 mm	Below 760 mm
Propane	—0·3°C	+ 0·3°C
Butane and butane-propane mixture	—0·4°C	+ 0·4°C

5.3 Report the value obtained in **5.2** to the nearest 0·1°C as '95 percent evaporated point at 760 mm of mercury'.

6. PRECISION

6.1 Results of duplicate tests shall not differ by more than the following amounts:

Type of LPG	*Repeatability*	*Reproducibility*
Butane and butane-propane mixture	0·5°C	1·0°C
Propane	0·5°C	1·3°C

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